

# BASIC STUDIES FOR THE ELECTRO POLISHING FACILITY AT DESY

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## Abstract

At DESY a TESLA test facility is operating since 1994. In this facility the cavity preparation bases on the application of buffered chemical polishing (BCP). From the results of the RF measurements it can be deduced that this preparation technique reaches a limitation at acceleration voltages of 30 MV/m. To exceed these values to the region of 40 MV/m an electro polishing system will be added to this facility (see this conference). In order to get the official permission ("Betriebs-genehmigung") by the German administration some basic information about the processes have to be studied. We report on measurements of samples which will give information on aging of the acid and the parameters like removal rates, polishing effect, out gassing of H<sub>2</sub> and O<sub>2</sub> correlated to this. On a test single cell an analysis of the amount and concentration of the gas development during the process will be studied. We report on parameter settings for the polishing process.

## 1. INTRODUCTION

At the Tesla Test Facility (TTF) nine cell superconducting resonators of 1,3 GHz resonance frequency undergo a preparation sequence of chemical treatment, high pressure rinsing and assembly in a class 10 clean room [Ref.1]. A total of about 120-200 µm of the niobium bulk material is etched away by a mixture of 1/1/2 HF/HNO<sub>3</sub>/H<sub>3</sub>PO<sub>4</sub> (1/1/2 BCP). Up to now a total of 70 cavities are prepared and tested in a vertical test insert. The static of the RF cold measurements shows that 30MV/m seems to be the maximum acceleration voltage to archived by BCP treatments [Ref.2].

In December of 2000 DESY decided to set up a facility for electro polishing (EP) of superconducting Cavities to transfer the encouraging results of EP single cell cavities to multi-cell resonators and to study the applicability for a large scale production like for TESLA [Ref.3]

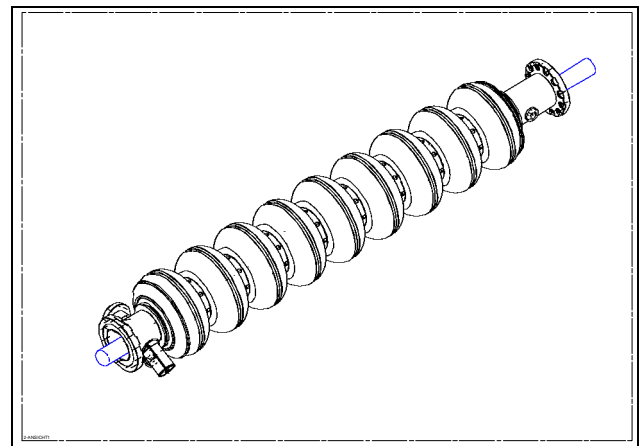
The baseline of the EP at DESY is the EP Mixture, developed by Siemens company in the 70's and further developed by KEK in collaboration with Namura Plating [Ref.4]. The acid in use is a mixture of 9 volume parts of sulphuric acid (96%) and 1 part fluoric acid (48%) at a temperature range of 30-40 °C. During the polishing process the cavity is orientated horizontally. The Cathode is made from pure aluminium which is covered by a polymeric net to reduce contact between niobium and H<sub>2</sub>.

The normal operating parameters for single cell cavities are well studied at CERN and KEK. In respect to a large scale production we have to answer the following questions. How does aging take affect on removal rates

and cavity smoothening or the area of oscillation of the amperage? What happens when the temperature is changing? To which level can the surface roughness of niobium be reduced?

## 2. CHEMICAL BASICS

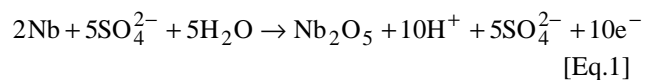
During the electro polishing process the niobium cavity works as the anode. The cathode is a tube made from pure aluminium, which is placed some millimeters off the axis of the beam tube (see Pic.1).



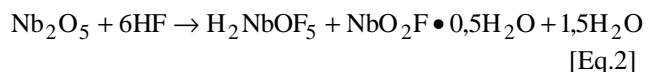
Pic.1: 9-cell-cavity with aluminium tube

By applying a voltage of 10-20 V the polishing process is activated. The current oxidizes the niobium surface first at the peaks because of the voltage enhancement. Hydrofluoric acid solutes these oxides. This process has a smoothening effect to the niobium surface. The electro chemical dissolution of the niobium anodes surface take place in three steps [Ref.5].

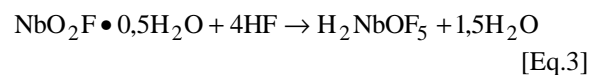
1<sup>st</sup>



2<sup>nd</sup>



3<sup>rd</sup>



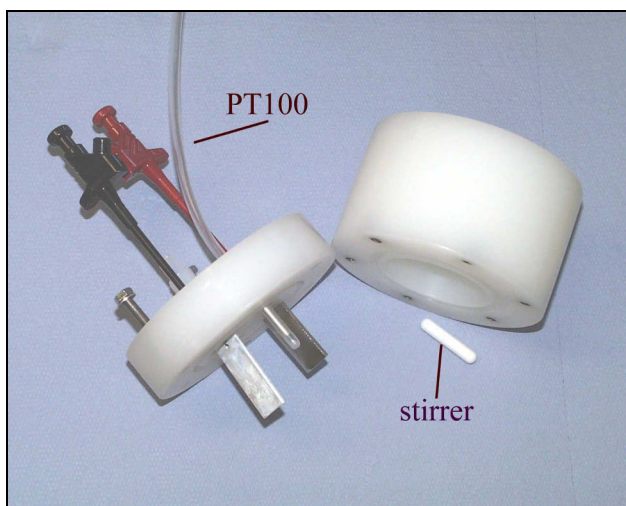
At the aluminium cathode  $H_2$  will be formed and gassed out. In addition an undesired electrolysis of water, which is a constituent part of the acid mixture, is possible.

### 3.ELECTRO POLISHING PARAMETERS

For our fundamental studies a sample size test apparatus is build. All experiments described in this paper are made with this apparatus

#### 3.1 Test apparatus

The test “reactor” is made of PVDF (polyvinyl-difluoride) and has a capacity of 200 ml. In the top flange (PVDF) four drill-holes are foreseen. Two serve as holders for the electrodes, one for the temperature sensor (PT100) and the forth one for the out gassing of  $H_2$  and  $O_2$ . To study the dependence of the polishing effect with the velocity of the acid the reactor is placed on the top of a magnetic stirrer (see Pic.2).



Pic.2: Test apparatus

Both electrodes are fixed at the bottom of the top flange by a clamp. The anode is made of a niobium sheet material and the cathode is made from pure aluminium sheet of 35 mm \* 20 mm \* 2,3 mm size. With a filling of 180 cm<sup>3</sup> acid a surface of 0.16 dm<sup>2</sup> (both sides of the electrode) is exposed to the acid and to the electro-chemical reaction.

#### 3.2 The influence of temperature and voltage

The aim of this experiment was to find out the dependence between the current density  $j[A/dm^2]$  and the changeable parameters of temperature  $T[^\circ C]$  and voltage. For this experiment we used fresh acid with 0g/l of niobium dissolved. At four different temperatures we altered the voltage until the point of the beginning oscillation is reached.

The result is an ascent of the temperature that causes the area of oscillation to begin at a lower voltage and a higher current density ( $j[A/dm^2]$ ) (see Fig.1).

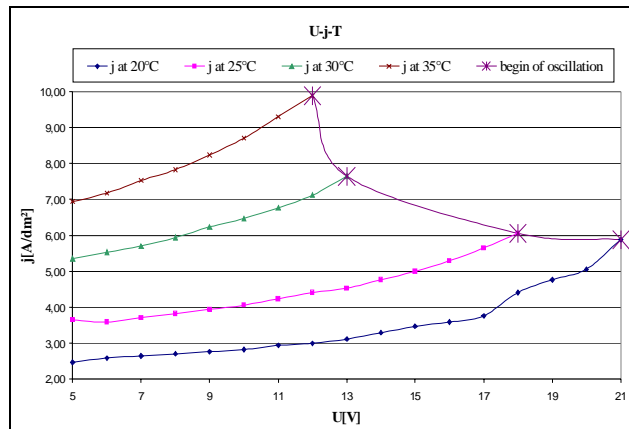


Figure 1: U-j-curves at different temperatures

The electrical resistance of the acid depends on the temperature. An ascent of the temperature causes an ascent of the ion mobility which implies a descent of the electrical resistance. For the voltage ohms law is valid.

#### 3.3 Aging effect

For a large scale production it is important to know how the properties of the acid mixture are changing when more and more niobium is dissolved. To determinate these changing parameters, we repeat the experiment (see 3.2) at three different loading grades of dissolved niobium. We used fresh acid, an acid with 4 g/l of niobium dissolved and an acid with 8 g/l of niobium dissolved.

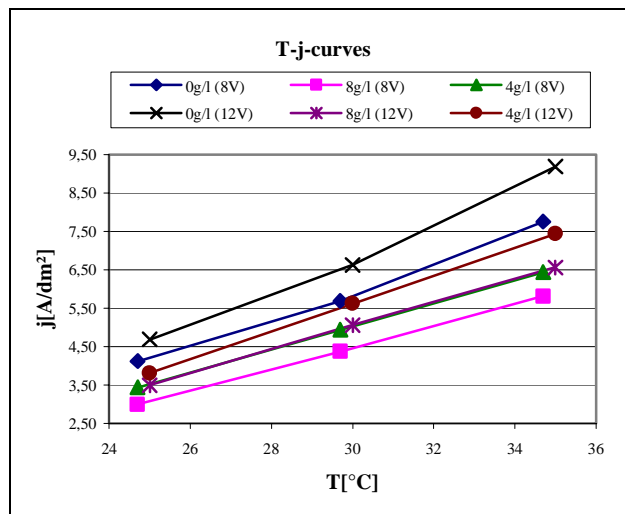


Figure 2: Aging effect (T-j-curves)

The result of this experiment shows (see Fig.2) that an ascending loading grade of niobium causing a descending current density at the same temperature. This allows the conclusion that the aging of the acid causes an ascent of the electrical resistance. A further conclusion is that the begin of the oscillation of an acid with 8g/l niobium

dissolved must be at a higher temperature then of a fresh acid, when the same voltage is used.

To proof this, we made another experiment with 2 different acids at a voltage of 15 V. The result confirms this conclusion (see Fig.3).

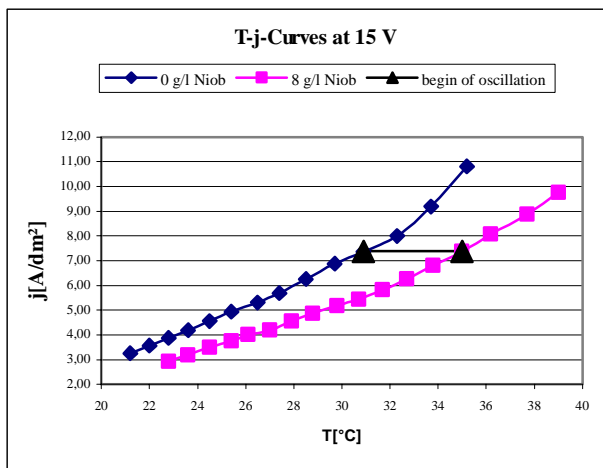
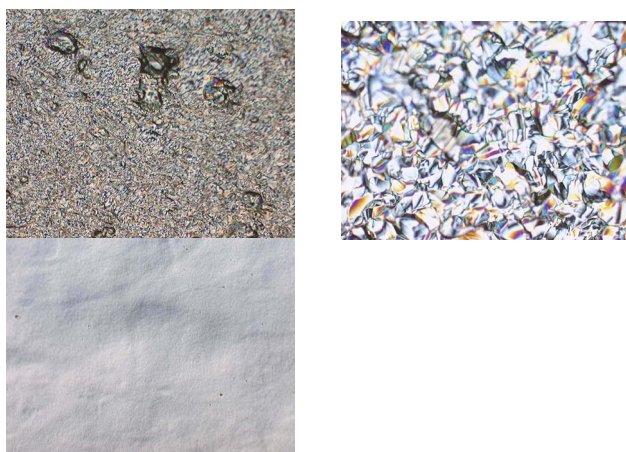


Figure 3: Aging effect (T-j-curves at 15 V) of the EP mixture

The aging of the acid effects the removal rates only indirectly. Old acid has the same removal rates per A/dm<sup>2</sup> as new acid. But at the same temperature and voltage is the current density of a new acid much higher then of an old acid. This effect cause a descent of the removal rates when the acid is aging.

#### 4. COMPARISON OF EP AND BCP SURFACE EFFECTS

To enhance the acceleration voltage to a region of 40 MV/m it is necessary to smoothen the niobium surface to a surface roughness of less then 0,5  $\mu\text{m}$  [Ref.6]. With the BCP-system this aim seemed to be impossible. The EP comes to this requirement. To proofs this.



Pic.3: Pictures in order raw sheet material, after 120 $\mu\text{m}$  BCP, after 120 $\mu\text{m}$  EP [Ref.7]

To proofs this two niobium material sheets were examined as raw material and after 120 $\mu\text{m}$  BCP respectively 120 $\mu\text{m}$  EP (see Pic.3). The measurements of the surface roughness definite.

For the raw material sheet we measured an average roughness of 0,6 $\mu\text{m}$ . After 120 $\mu\text{m}$  BCP the average roughness was 1,5 $\mu\text{m}$  with a max. difference of 7,7 $\mu\text{m}$  at a measuring length of 4mm. The average roughness after 120 $\mu\text{m}$  EP was 0,2 $\mu\text{m}$  with a max. difference of 1,5 $\mu\text{m}$ .

#### 5.OBSERVATIONS

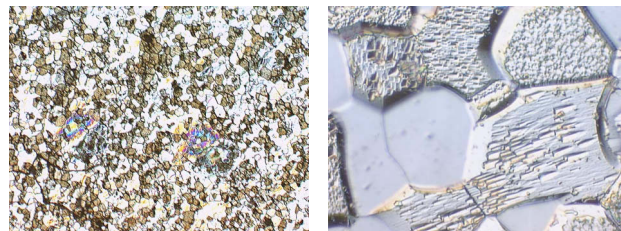
During the experiments we made three observations about the behavior of the EP acid mixture.

##### 5.1 Another area of oscillation

At a low voltage (3-7 V) is another area of oscillation. Today we have no idea what effect this oscillation causes.

##### 5.2 Etching pits effect

After the EP was finished, the warm acid (about 40°C) was not removed from test reactor for two hours. After that time the Niobium surface shows a partially roughness (see Pic.4)



Pic.4: Niobium with the etching pits effect [Ref.7]

We repeated this experiment a few times with the same result. The etching pits effect happen in two of three experiments.

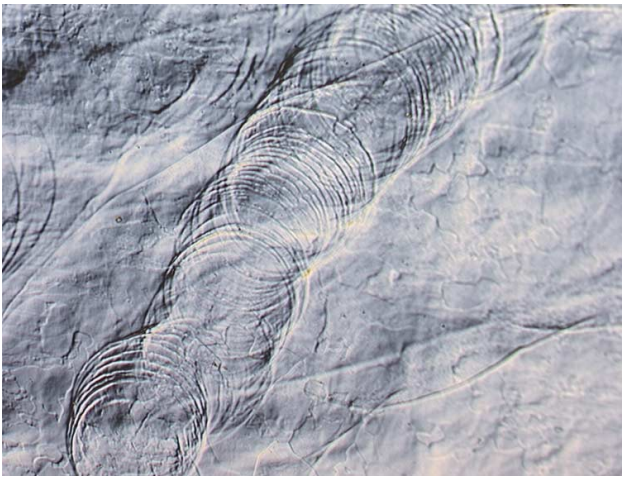
##### 5.3 Bubble traces on the polished surface

During the EP it could happen, that gas bubbles grow on the niobium surface. These bubbles will leave traces on the surface (see Pic. 5/6).

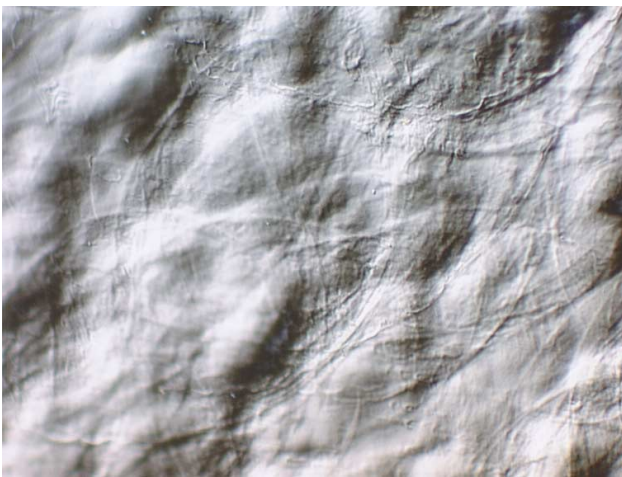
The cathode (were H<sub>2</sub> is formed) is coated with a polymeric net to minimize this effect.

If the voltage is too high the hydrolysis of H<sub>2</sub>O will start. In that case O<sub>2</sub> was formed at the niobium surface which has the same effect.





Pic. 5: Bubble traces [Ref.7]



Pic. 6: Bubble traces [Ref.7]

## 6. REFERENCES

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